CHAPTER 3

METHODOLOGY

3.1 INTRODUCTION

In this chapter, the copper ferrites ($CuFe_2O_4$) preparation procedure is described followed by the characterization of the photocatalyst. The characterization techniques employed in the current work were nitrogen (N_2) physisorption for textural property determination, UV-vis DRS scanning for band gap energy determination, field emission scanning electron microscopy-energy dispersive X-ray (FESEM-EDX) for surface morphology capturing and element determinations, X-ray diffraction (XRD) technique for obtaining crystalline structure diffraction pattern and particle size analyser to determine the particle size distribution. The methods of photoreaction glycerol degradation, sample analysis, effects of reaction parameters and factorial analysis by Design Expert are presented too.

3.2 MATERIALS

3.2.1 Chemicals

The chemicals needed in this study are listed in the Table 3.1. Apart from the ultrapure water, all the chemicals listed in Table 3.1 were procured either from Sigma-Aldrich, Merck or Fischer Chemical. All these chemicals were used without further

purification. The ultrapure water was obtained from the Millipore Elix 5-UV unit which is readily available in the laboratory of Universiti Malaysia Pahang (UMP). Ultrapure water was used for the catalyst preparation and preparation of different concentrations of reactants.

3.2.2 Gases

The gases required in the study are listed in Table 3.2. All gases used in this research were supplied by MOX-Linde Gases Sdn. Bhd. Table 3.2 also lists the purity of the gases and their application in this study.

Table 3.1: List of chemicals

Chemical	Purity	Brands	Application
Copper (II) nitrate trihydrate $Cu(NO_3)_2.3H_2O$	> 98%	Sigma-Aldrich	Catalyst preparation
Iron (III) nitrate nonahydrate $Fe(NO_3)_3.9H_2O$	> 98%	Sigma-Aldrich	Catalyst preparation
Citric acid ($C_6H_8O_7$)	≥ 99.5%	Sigma-Aldrich	Catalyst preparation
Glycerol ($C_3H_8O_3$)	≥ 99%	Sigma-Aldrich	Photoreaction
Hydrogen peroxide (H_2O_2)	30%	Merck	Photoreaction
Acetonitrile (C_2H_3N)	HPLC grade	Fisher Chemical	Mobile phase HPLC

Table 3.2: Lists of gases

Gases	Purity	Application
N_2/He mixture	N_2=30.03%, He=balance	N_2 physisorption
He	> 99.996%	N_2 physisorption
N_2	> 99.99%	N_2 physisorption

3.3 PHOTOCATALYST PREPARATION

The photocatalyst used in the present study was $CuFe_2O_4$. It was synthesized via sol–gel method adapted from previous works (Shen et al., 2013; Yang et al., 2009). Firstly, 0.005 mol $Cu(NO_3)_2$ and 0.010 mol $Fe(NO_3)_3$ were co-dissolved in 50 ml distilled water. The mixed solution was subsequently added into 100 ml of 0.3 M citric acid solution. This step would produce a transparent mixed sol. During this mixing procedure, the temperature was controlled at around 80°C until transparent and viscous gel was obtained. The as-synthesized gel was subsequently transferred into an oven and kept at 140°C for 3 h. The dried photocatalyst was then air-calcined at 850°C for 3 h employing a ramping rate of 10°C min^{-1}. The calcined catalyst was then ground for catalyst characterization and photocatalytic Fenton study.

3.4 CATALYST CHARACTERIZATION

3.4.1 N_2 Physisorption

The specific surface area of photocatalyst was determined by N_2 physisorption. The most commonly employed equation is the Brunauer-Emmett-Teller (BET) model. The BET equation (Brunauer et al., 1938) is derived for multilayer adsorption and based on the relationship between the volume of gas physically adsorbed and the total area of adsorbent as shown in Eq. (3.1):

$$\frac{\mathrm{P}}{\mathrm{V}(\mathrm{P_s}-\mathrm{P})} = \frac{1}{cV_m} + \frac{(c-1)\mathrm{P}}{cV_m\mathrm{P_s}} \tag{3.1}$$

where

P = gas pressure (Pa)

P_s = saturation pressure of the adsorbate gas (Pa)

V = volume of gas adsorbed (mL)